JP 10-088,472

Job No.: 1505-96760 Ref.: JP10088472
Translated from Japanese by the Ralph McElroy Translation Company
910 West Avenue, Austin, Texas 78701 USA

JAPANESE PATENT OFFICE
PATENT JOURNAL (A)
KOKAI PATENT APPLICATION NO. HEI 10[1998]-88472

| | | |
|---|---|---|
| Int. Cl.[6]: | D 06 L | 3/02 |
| | C 11 D | 7/18 |
| | | 7/42 |
| | C 12 S | 3/04 |
| | D 01 C | 1/00 |
| | D 01 C | 1/02 |
| | D 06 L | 3/10 |
| | //D 06 M | 16/00 |
| | D 06 M | 16/00 |

Filing No.: Hei 8[1996]-257776

Filing Date: September 6, 1996

Publication Date: April 7, 1998

No. of Claims: 1 (Total of 8 pages; FD)

Examination Request: Not filed

# METHOD FOR SCOURING AND BLEACHING OF NATURAL CELLULOSE TYPE FIBERS

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[There are no amendments to this patent.]

Abstract

Objective

The objective of the present invention is to provide a method for scouring and bleaching of cotton fiber or linen fiber based on a combined use of a pectin-decomposing enzyme and hydrogen peroxide.

Means of solution

In a method for scouring of natural cellulose type fiber and/or fiber containing a natural cellulose type fiber and workpieces made of the same using an enzyme composition containing a pectin-decomposing enzyme, a method for scouring and bleaching of natural cellulose type fiber and/or fiber containing a natural cellulose type fiber and workpieces made of the same characterized by the fact that an oxidizing agent and a reducing agent are simultaneously used.

Claim

1. A method for scouring of a natural cellulose type fiber and/or a fiber containing a natural cellulose type fiber and workpieces made of the same using an enzyme composition containing a pectin-decomposing enzyme, which method for scouring and bleaching of natural cellulose type fiber and/or fiber containing a natural cellulose type fiber and workpieces made of the same is characterized by the fact that an oxidizing agent and a reducing agent are simultaneously used.

Detailed explanation of the invention

[0001]

Industrial application field

The present invention pertains to a method for scouring and bleaching of natural cellulose type fibers. More precisely, the invention pertains to a method for scouring and bleaching of cotton fiber or linen fiber based on the combined use of a pectin-decomposing enzyme and hydrogen peroxide.

[0002]

Prior art

Primary natural foreign matter and secondary foreign matter imparted during a spinning process, spinning process [sic], weaving process, and knitting process are included in woven or knitted materials made of natural fibers and synthetic fibers. Currently, the aforementioned unwanted foreign matter is removed through a desizing process, scouring process, bleach process, etc. to take full advantage of the basic properties of a variety of fibers and to facilitate the dye finishing process. Furthermore, the method used in the aforementioned processes varies depending on the type of fiber.

[0003]

For example, in the scouring process for cotton fiber, the treatment is done at high temperature using sodium hydroxide and a surfactant since the cotton fiber includes natural foreign matter such as pectin and cotton wax. In other words, when a boiling treatment in sodium hydroxide is provided, the pectin is converted to water-soluble sodium pectinate or its molecular weight is reduced and it becomes soluble in water and is removed. Furthermore, cotton wax and fats and oils undergo saponification or hydrolysis in the presence of strong alkali and efficient removal can be achieved when a surfactant is used in combination.

[0004]

In a bleaching process, colored materials included in the fiber, yarn, or cloth are decomposed and whiteness is increased.

[0005]

Furthermore, unlike a treatment based on chemical products, treatment methods for a variety of fibers with enzymes have been known in the past. For example, for scouring of silk products, bacterial protease is used, for removal of protein impurities in sheep's wool, protease is used and for removal of mutton tallow, lipase is being examined.

[0006]

And in recent years, use of enzymes in a scouring process for cotton has been proposed. In other words, a scouring method for cotton fibers where acidic pectinase and alkali pectinase (hereinafter referred to as pectinase), commercially available for processing food items, are used has been reported (Fiber Soft With Respect to Humans and Its Processing, pp. 300-303, published by Fiber Co., Ltd., August 1995).

[0007]

Problems to be solved by the invention

In conventional scouring methods for fibers where chemical products such as sodium hydroxide are used, large amounts of chemicals are used and from the standpoint of environmental protection, a reduction or elimination of the chemicals is desired. And for this reason, the above-mentioned scouring method that utilizes enzymes is gaining attention to eliminate environmental problems.

[0008]

Furthermore, a bleaching process is provided for natural cellulose type fibers, as needed, and in conventional methods where a chemical such as sodium hydroxide is used [for scouring], washing of the fibers after scouring is required and bleaching with a bleach such as hydrogen peroxide is done independently.

[0009]

Means to solve the problem

In scouring of natural cellulose type fibers with an enzyme, the scouring and bleaching of the natural cellulose type fibers can be simplified when bleaching can be simultaneously done. Based on the above background, and as a result of much research conducted by the present inventors in an effort to eliminate the above-mentioned existing problems, bleaching can now be simultaneously done in a method where scouring of natural cellulose type fibers is done with an enzyme, and with this result, the present invention was accomplished.

[0010]

In other words, the present invention is a scouring and bleaching process for natural cellulose type fibers characterized by the fact that bleaching is simultaneously done in a scouring method for natural cellulose type fibers with an enzyme.

[0011]

The above-mentioned pectinase may be used in this invention as the enzyme for scouring of natural cellulose type fibers, but it is further desirable when an enzyme produced by a microbe newly obtained by the present inventors by screening from soil is used.

[0012]

It should be noted that each enzyme activity is shown by measurement values obtained by the methods described below unless otherwise specified.

[0013]

① Pectin-decomposing activity

Mixing is performed for 1 mL of 0.5% pectin aqueous solution, 0.08 mL of 100mM EDTA aqueous solution, and 0.72 mL of 0.2M phosphoric acid buffer solution (pH 8). Furthermore, 0.2 mL of enzyme solution is added and a reaction is conducted for 10 min at 30°C. Furthermore, 2 mL of 0.2M acetic acid buffer solution (pH 4.5) is added to terminate the reaction and absorption at a wavelength of 235 nm is measured. The amount of enzyme that produces 1 μmol of unsaturated galacturonic acid per 1 min is defined as 1 unit. In this case, the molecular absorption coefficient of unsaturated galacturonic acid is 4600.

[0014]

② Pectic acid decomposing activity

Mixing is done for 1 mL of 0.5% pectic acid aqueous solution, 0.7 mL of acid buffer solution (pH 8) and 1 mL of 10mM calcium chloride solution; then, 0.2 mL of enzyme solution is added and a reaction is conducted for 10 min at 30°C. Furthermore, 2 mL of 0.2M acetic acid buffer solution (pH 4.5) are added to terminate the reaction and absorption at a wavelength of 235 nm is measured. The amount of enzyme that produces 1 μmol of unsaturated galacturonic acid per 1 min is defined as 1 unit. In this case, the molecular absorption coefficient of unsaturated galacturonic acid is 4600.

[0015]

③ Fiber decomposing activity (CMCase)

1 mL of enzyme solution is added to 4 mL of 0.5% carboxymethylcellulose solution (pH 8.0) and a reaction is conducted for 30 min at 40°C. 2 mL of Somogyi [transliteration] solution are added to terminate the reaction, and heating is done for 20 min at 100°C. After cooling, 1 mL of arsenic ammonium molybdate solution is added and cuprous oxide is dissolved, then, water is added to make 25 mL and absorption at a wavelength of 500 nm is measured. The amount of enzyme that produces a reducing agent, etc. corresponding to 1 mg of glucose in 1 min is defined as 100 units.

[0016]

④ Fiber material decomposition activity ($C_1$ase)

The rotational speed for a mono (Monod) type vibrating constant temperature bath is set at 60 rpm and the amplitude at 7 cm, then, 5 mL of enzyme solution (pH 8.0) are poured into an L type test tube and stored for 5 min at 40°C. Furthermore, 2 pieces of filter paper (Toyo Filter

Paper, No. 51) with a size of 1 cm x 1 cm are added and reaction is conducted under vibration. The time (min) required for complete disintegration of the two pieces of paper is measured. The mean value of 7 measurement results eliminating the highest value and the lowest value is calculated. The amount of enzyme capable of achieving complete disintegration of the filter paper in 1 min is defined as 10,000 units.

[0017]

The invention is explained in further detail below. The present inventors searched for a supply source in nature to obtain microbes for production of an enzyme effective for pectin and found that a strain separated from soil produces an enzyme effective for novel pectin.

[0018]

Identification of the mycological characteristics of the strain separated by the present inventors was done using references ① to ⑤ below.

[0019]

References cited: ① Lelliott, R.A. 1974. Genus XII. *Erwinia* Winslow et al. 1920, 209. p. 332-340. In R.E. Buchanan and N.E. Gibbons (ed.), Bergey's manual of determinative bacteriology, 8th ed. The Williams & Wilkins Co., Baltimore.

[0020]

② Lelliott, R.A. and Robert S. Dickey. 1984. Genus VII. *Erwinia* Winslow et. al. 1920, 209AL. p. 469-476. In N.R. Krieg and J.G. Holt (ed.), Bergey's manual of systematic bacteriology, vol. 1. The Williams & Wilkins Co., Baltimore.

[0021]

③ Goto, M., K. Matsumoto. 1987. *Erwinia carotovora* subsp. *wasabiae* subsp. nov. Isolated from diseased rhizomes and fibrous roots of Japanese horseradish (Eutrema wasabi Maxim.). Int. J. Syst. Bacteriol. 37: 130-135.

[0022]

④ Gallois, A. et al. 1992. *Erwinia carotovora* subsp. *odorifera* susbsp. nov., Associated with Odorous soft rot of chicory (Chichoriumintybus L.) Int. J. Syst. Bacteriol. 42: 582-588.

[0023]

⑤ Alcorn, S.M. et al. 1991, Taxonomy and pathogenicity of *Erwiniacacticida*_sp. nov. Int. J. Syst. Bacteriol. 41: 197-212.

Mycological characteristics of the aforementioned microbe are listed below.

[0024]

(A) Morphological characteristics

Shape and size of the cell: Bacillus, 1.5 to 2.4 μ x 0.5 to 0.7 μ, seldom reaches a length of 10 μ.

Polymorphism of cell: None

Mobility: None

Spores: Not formed

Gram's stain: Negative

[0025]

(B) Incubation nature

Broth agar plate incubation: round, smooth, entire, circular convex, semi-transparent, wet light, colorless (common agar culture medium 'Eiken')

Litmus milk incubation: weak acidity, undergoes coagulation, forms pepton, reduces litmus, non-gas forming

[0026]

(C) Physiological characteristics

(1) Condition for oxygen: facultative anaerobic

(2) Dye formation

Pink colored water-soluble dye: negative

Blue colored insoluble dye: negative

Yellow colored insoluble dye: negative

(3) Mucoid-like growth and development: negative

(4) Growth and development temperature: excellent growth at 20 to 34°C. Slight growth and development at 37°C but no growth and development observed at 40°C.

(5) Formation of hydrogen sulfide from cysteine: Positive (lead acetate test paper method)

(6) Formation of reduced form material from sucrose: positive

(7) MR test: negative

(8) VP test: positive

(9) Urease: negative
(10) Decomposition of pectin: positive
(11) Gas formation from glucose: negative
(12) Decomposition of casein: positive
(13) Decomposition of gelatin: positive
(14) Phenylalanine deaminase: negative
(15) Arginine dihydrase: negative
(16) Lysin decarboxylase: negative
(17) Ornithine decarboxylase: negative
(18) Indole formation: negative
(19) Nitrate reducibility: positive
(20) Growth and development in 5% sodium chloride: positive
(21) Deoxyribonuclease: positive
(22) Phosphatase: positive
(23) Lecithinase: negative
(24) Hydrolysis of starch: negative
(25) Use of citrate: positive
(26) Use of Malonate: negative
(27) Oxytase: negative
(28) Catalase: positive
(29) OF test: Fermentation
(30) Decomposition of esculin: positive
(31) Decomposition of Tween 80: positive
(32) Acid formation
Melibiose: +
Lactose: +
Inositol: -
Rhamnose: +
Raffinose: +
Esclin: +
Inulin: +
Salicin: +
Soluble starch: -
Xylose: +
Maltose: -
Trehalose: -

L-arabinose: -

Dulcitol: -

Sorbitol: +

Glycerol: -

Ribose: +

Adonitol: -

Mannose: +

Melezitose: -

Mannitol: +

α-methyl-D-Glucoxide: +

Cellobiose: +

[0027]

Since the present strain is facultative anaerobic, catalase positive, oxytase negative, decomposes sucrose through fermentation, non-gas-forming from glucose, urease negative, phenylalanine deaminase negative, VP test positive, MR test negative, nitrate reducibility positive and the optimum formation temperature is not 37°C, the strain is classified as Erwinia (Erwinia) genus (Reference ① and ②).

[0028]

For strains capable of decomposing pectin among Erwinia genus, *E. carotovora subsp. carotovora, E. caratovora subsp. atroseptica, E. carotovora subsp. betavasculorum, E. carotovora subsp. odorifera, E. carotovora subsp. wasabiae, E. rubrifaciens, E. salicis, E. chrysanthemi, E. cacticida*, can be mentioned. However, some of characteristics of the present microbe are different from those of the aforementioned genus and a corresponding genus does not exist.

[0029]

Table 1

| ① 試験項目 | A | B | C | D | E | F | G | H | I | J |
|---|---|---|---|---|---|---|---|---|---|---|
| ② 36℃での生育 | + | + | − | + | − | + | + | − | + | + |
| ③ シュクロースから還元性物質 | + | − | + | + | − | + | − | + | − | − |
| インドールの生成④ | − | − | − | − | − | − | − | − | + | − |
| ⑤ マロン酸塩の利用 | − | − | − | − | − | − | − | − | + | + |
| 酸：メリビオース⑥ | + | + | + | − | − | + | − | + | + | − |
| ⑦ マルトース | − | − | + | + | − | d | − | − | − | − |
| L-アラビノース⑧ | − | + | + | ND | ND | + | + | − | + | − |
| ⑨ ラクトース | + | + | + | + | + | + | − | − | d | d |
| トレハロース1) | − | + | + | + | + | + | − | − | − | d |
| 1! α-メチル-D-グルコシド | + | − | + | + | − | + | + | − | − | − |

Key:
1 Test item
2 Growth and development at 36°C
3 Formation of reduced form material from sucrose
4 Formation of indole
5 Use of malonate
6 Acid: Melibiose
7 Maltose
8 L-arabinose
9 Lactose
10 Trehalose
11 α-methyl-D-Glucoxide

[0030]

A: Erwinia sp No. 9482
B: E. carotovora subsp. carotovora (quoted from reference ① and ②)
C: E. carotovora subsp. atroseptica ( 〃 ①,② 〃 )
D: E. carotovora subsp. betavasculorum ( 〃 ③ 〃 )
E: E. carotovora subsp. wasabiae ( 〃 ③ 〃 )
F: E. carotovora subsp. odorifera ( 〃 ④ 〃 )
G: E. rubrifaciens ( 〃 ①,② 〃 )
H: E. salicis ( 〃 ①,② 〃 )
I: E. chrysanthemi ( 〃 ①,② 〃 )
J: E. cacticida ( 〃 ⑤ 〃 )

[0031]

In Table 1, d indicates that 11 to 89% of stock is positive, and ND is absence of data.

[0032]

As described above, the present microbe does not match any existing type, thus, it is recognized as a new species of Erwinia (Erwinia) genus and named as Erwinia sp (Erwinia sp.) No. 9482.

[0033]

Furthermore, Erwinia sp No. 9482 is deposited as FERM P-15696 in the Industrial Technical Institute, Bioengineering Division, Agency of Industrial Science and Technology of the Ministry of International Trade and Industry.

[0034]

For the incubation method used for the present strain, in general, a liquid culture can be used. For an example of liquid culture, the method shown below can be mentioned. The culture component used is not especially limited and those commonly used for incubation of microbes may be used in this case as well.

[0035]

For example, for a carbon source, saccharides such as glucose, sucrose, sorbitol, fructose, glycerol, dextrin, molasses, and starch hydrolysates, organic acids such as acetic acid and fumaric acid, etc. may be used.

[0036]

For a nitrogen source, pepton, enzyme extract, corn steep liquor, casein hydrolysate, broth extract, nitrates, ammonium salts, broth extract [sic], enzyme powder, soybean hydrolysate, cotton seed powder, benthon, etc. can be mentioned.

[0037]

Furthermore, those added with inorganic salts such as potassium salt, magnesium salt, sodium salt, phosphate, manganese salt, iron salt, and zinc salt may be used.

[0038]

Incubation is done at an incubating temperature in the range of 10 to 50°C, preferably, 25 to 40°C, a pH in the range of 3 to 9, preferably, 5 to 7, and in general, for 10 to 30 h under aerobic conditions. For example, a vibration incubation method, aerobic deep incubation method with jar fermentor, etc. may be used.

[0039]

A culture material containing crude enzyme can be obtained from the culture filtrate after removing the biomass from the culture material produced. The enzyme activity of the culture material produced has been measured.

[0040]

The enzyme composition used in the present invention can be utilized as the culture solution where incubation of the above-mentioned microbe is done.

[0041]

Needless to say, an adjustment of the enzyme concentration is possible according to the reaction conditions used for scouring the natural cellulose type fibers. Furthermore, the enzyme composition is essentially absent for fiber element decomposition activity.

[0042]

In the following, the scouring method for fibers using the above-mentioned enzyme composition is described. The target fiber is a fiber containing pectin as a component and is a natural cellulose type fiber, and/or a fiber containing natural cellulose type fibers and workpieces of the same. Furthermore, for natural cellulose type fibers, cotton and linen can be mentioned. For linens, ramie (ramie), linen (flax), jute, jute (jute) [sic], Manila hemp and sisal linen can be mentioned.

[0043]

For example, fibers containing non-processed natural cellulose type fibers and natural cellulose type fibers, workpieces such as woven materials, knitted materials, non-woven fabrics, yarn, cotton of the same can be mentioned, the aforementioned materials are soaked in a solution of the above-mentioned enzyme composition, and the processing conditions vary depending on the concentration of the enzyme, its purity, the working temperature, the working pH, etc.

[0044]

Especially when the enzyme composition produced by the above-mentioned microbe is used, the amount used can be reduced significantly in comparison to a conventional case where many different types of pectin active enzymes are used because of its wide pH range, etc. For example, a pH in the range of 6 to 11 may be used, and the temperature used is in the range of 20 to 60°C and the time is in the range of approximately 30 min to 24 h.

[0045]

Furthermore, a pre-treatment such as desizing may be provided before the scouring treatment, or said treatment may be simultaneously done.

[0046]

Furthermore, a bleaching process may be performed at the time of the aforementioned scouring process. Many different types of oxidizing agents and reducing agents may be used for the bleaching process. For examples of common oxidizing agents, hydrogen peroxide, peracetate, sodium nitrite, sodium hypochlorite, and calcium hypochlorite can be mentioned, and hydrogen peroxide is especially suitable for use. Furthermore, for common reducing agents, hydrosulfite, sodium sulfite, thiourea dioxide, etc. can be mentioned.

[0047]

The present invention is explained in further detail with application examples and comparative examples below, but the present invention is not limited to the application examples shown below.

[0048]

Application examples

Application Example 1

Incubation of Erwinia sp No. 9482 (FERM P-15696) was done for 32 h in 500 L of culture medium containing 0.3% pectin, 0.3% ammonium sulfate, 0.1% potassium diphosphate, 1.5% lactose, 2% pepton, 0.5% enzyme extract, and 0.1% calcium carbonate at 28°C, at an airflow of 100 L/min, and at a rotational speed of 200 rpm. Centrifugal separation was done to remove the biomass from the culture solution so as to produce a crude enzyme solution. The pectic acid decomposition activity, pectin decomposition activity, fiber decomposition activity, and fiber element decomposition activity of the aforementioned enzyme solution were measured.

[0049]

As a result, the pectic acid decomposition activity was 24 units/mL, and the pectic acid decomposition activity : pectin decomposition activity : fiber decomposition activity : fiber element decomposition activity was 1:0.14:8.3:0. In other words, the pectic acid decomposition activity : pectin decomposition activity = 1:0.14 and the pectic acid decomposition activity : fiber decomposition activity = 1:8.3.

[0050]

Application Example 2

1 g of cotton cloth was placed in a 20 mL solution of 40mM Briton-Robinson buffer solution (pH 10) containing 0.01% Cintol [transliteration] CO-300 (surfactant) and 1% of 30% hydrogen peroxide, then, the enzyme solution (0.08 units added with respect to pectic acid decomposing activity) produced in Application Example 1 was added and a reaction was conducted at 40°C for 30 min; treatment was done at 90°C for 15 min and the enzyme was deactivated. The whiteness of the cotton cloth after the reaction was measured according to the method described below. Furthermore, the free pectin was measured by the method described below and the result obtained is shown in Table 1 [sic; 2]. For comparison, the case of a conventional alkali agent treated material and the case where hydrogen peroxide was omitted were used.

[0051]

Carbazole sulfuric acid method

For 0.25 mL of sample, 0.25 mL of an ethanol solution containing 0.2% carbazole and 3 mL of 84% sulfuric acid solution were added and vigorous stirring was provided. The solution was then left standing for 20 min at 75°C, and after cooling, absorption at a wavelength of 525 nm was measured. Meanwhile, a calibration curve was made with D-α-galacturonic acid, and the amount of pectin was obtained from the aforementioned calibration curve.

[0052]

Table 2

| ① 条　件 | ② 遊離したペクチン量 mg／綿1g | ③ 白色度 |
|---|---|---|
| ④ 酵素処理前 | | 81．1 |
| ⑤ アルカリ剤処理 | 7．3（100%） | 89．4 |
| ⑥ 過酸化水素無添加 | 7．0（95.9%） | 81．1 |
| ⑦ 過酸化水素　添加 | 6．8（93.2%） | 90．1 |

Key: 1 Condition
2 Amount of free pectin mg/1 g gossypium
3 Whiteness degree
4 Before enzyme treatment
5 Alkali agent treatment
6 Without hydrogen peroxide
7 With hydrogen peroxide

[0053]

The numbers inside the ( ) are values with the alkali-treated case at 100. As is clearly shown in Table 2, the removal of pectin is not affected as a result of the addition of hydrogen peroxide and a good bleaching effect was achieved.

[0054]

Application Example 3

1 g of cotton cloth before desizing was placed in a 20 mL solution of 40mM Briton-Robinson buffer solution (pH 7) containing 0.01% Cintol CO-300 (surfactant), 1% of 30% hydrogen peroxide, and 0.01% Amilase [transliteration] AD (product of Amano Pharmaceuticals), then, the enzyme solution (0.08 units added with respect to pectic acid decomposing activity) produced in Application Example 1 was added and a reaction was conducted at 40°C for 30 min; treatment was done at 90°C for 15 min and the enzyme was deactivated. The whiteness of the cotton cloth after the reaction was measured. Furthermore, the free pectin was measured and the results obtained are shown in Table 3. Furthermore, the iodine starch reaction was used to confirm adequate desizing.

[0055]

Table 3

| ① 条　件 | 遊離したペクチン量 ㎎／約1ｇ ② | 白色度 ③ |
|---|---|---|
| ④ 酵素処理前 | | 80.2 |
| ⑤ アルカリ剤処理 | 7.5 (100%) | 89.8 |
| ⑥ 同時処理 | 7.1 (94.7%) | 86.5 |

Key: 1 Condition
2 Amount of free pectin mg/1 g gossypium
3 Whiteness degree
4 Before enzyme treatment
5 Alkali agent treatment
6 Simultaneous treatment

[0056]

The numbers inside the ( ) are the values with the alkali-treated case at 100. As is clearly shown in Table 3, sizing, scouring and bleaching can be simultaneously done.

[0057]

Application Example 4

1 g of linen fiber (warp: linen 40%, ramie 60%, density 42 fibers/inch, cotton yarn number count 9S, weft: linen 50%, ramie 60% [sic], density 42 fibers/inch, cotton yarn number count 9S, plain weave) before desizing was placed in a 20 mL solution of 20mM sodium phosphate buffer solution (pH 8) containing 1, 1.5 and 2% each of 0.2% Liponox [transliteration] NCI (product of Lion Co., nonionic surfactant), 0.01% Amilase AD (product of Amano Pharmaceuticals), and 35% hydrogen peroxide solution, respectively; then, the enzyme solution (0.08 units added with respect to pectic acid decomposing activity) produced in Application Example 1 was added. Then, a reaction was conducted at 40°C for 30 min; treatment was performed at 95°C for 30 min and deactivation of the enzyme was achieved. The whiteness of the linen fiber after drying and the amount of residual pectin were measured according to the methods described below. And the results obtained are shown in Table 4.

[0058]

Table 4

| ① 条　件 | ② ペクチン残存量 K/S | ③ 白色度 |
|---|---|---|
| ④ 処理前 | 5．08 | 64．6 |
| ⑤ 過酸化水素　0% | 2．29 | 65．9 |
| 過酸化水素　1% | 2．24 | 89．1 |
| 過酸化水素 1.5% | 2．65 | 90．3 |
| 過酸化水素　2% | 2．74 | 91．0 |

Key: 1 Condition
2 Amount of residual pectin K/S
3 Whiteness degree
4 Before treatment
5 Hydrogen peroxide

[0059]

Adequate removal of pectin was possible even when hydrogen peroxide was used and whiteness based on visual examination showed a practical level of bleaching effect.

[0060]

Application Example 5

10 kg of cotton yarn (40 yarn number count, two-ply yarn) were dipped in 180 L of scouring and bleaching solution comprised of the enzyme solution produced in Application

Example 1, 8.3 mL/L of 35% hydrogen peroxide solution, 2 g/L of Liponox NCI (product of Lion Co., nonionic surfactant), and 0.37 g/L of sodium carbonate and having a pectic acid decomposing activity of the enzyme of 2.9 μ/mL, and a reaction was performed for 20 min at 40°C using a hank dyeing machine. Then, treatment was conducted at 95°C for 10 min and the enzyme was deactivated.

[0061]

Application Example 6

5.5 mL/L of 35% hydrogen peroxide and 0.32 g/L of sodium carbonate were used and deactivation of the enzyme was done at 95°C for 20 min. And the rest of the treatment was done under the same conditions described in Application Example 4.

[0062]

Comparative Example 1

The cotton yarn used in Application Example 5 was dipped in 180 L of scouring and bleaching solution comprised of 4.4 mL/L of 35% hydrogen peroxide, 1.1 g/L of Gran-Up [transliteration] V0-50K (product of Sanyo Kasei Co., scouring coagent) and 2 g/L of sodium hydroxide and scouring and bleaching were done at 95°C for 30 min using a hank dyeing machine as described in Application Example 5. The whiteness degree, single yarn strength, residual pectin of the cotton yarn provided with scouring and bleaching in the above-mentioned Application Example 5, Application Example 6, and Comparative Example 1 are shown in Table 5 below.

[0063]

Table 5

| | 綿糸の性状 ① | | |
|---|---|---|---|
| | ② 白色度 | 単糸強力(g/f) 平均 ③ ±SD(n=20) | ペクチン 残存量 (K/S) ④ |
| ⑤ 実施例5 | 90.4 | 526.5 ±34.1 | 1.78 |
| ⑤ 実施例6 | 90.0 | 499.0 ±25.5 | 1.80 |
| ⑥ 比較例1 (従来法) | 89.0 | 520.0 ±20.0 | 0.70 |

Key: 1 Properties of cotton yarn
2 Whiteness degree
3 Single yarn strength (g/f) average
4 Pectin residual ratio (K/S)
5 Application Example ___
6 Comparative Example 1
(Conventional method)

[0064]

In this case, each measurement method used in Table 5 is as shown below.

[0065]

① Whiteness of yarn

The yarn was wrapped around a black bobbin winding of 6 x 6 $cm^2$ alternatingly in the vertical direction and horizontal direction at least 6 times to produce a sample. Production of the sample was based on the method specified in JIS L 1095 (Standard woven material testing method). For four given points on the surface of the sample, L, a and b values are measured using a CR-200 color difference meter (product of Minolta Camera Co.) and the whiteness was determined based on the values of L, a and b according to the Hunter method using the equation below.

[0066]

Equation 1

$$\text{Whiteness} = 100 - \{(100 - L)^2 + a^2 + b^2\}^{1/2}$$

[0067]

② Single yarn strength

The measurement method is based on the method specified in JIS L 1095 (single yarn strength).

[0068]

③ Residual pectin in yarn

The yarn is soaked in 0.02% ruthenium red solution at a bath ratio of 1:40 and vibration is performed for 10 min at 30°C. After vibration, a water wash is performed, vibration is further performed for 30 min at 50°C and a hot-water wash is provided followed by drying. The aforementioned yarn was wrapped around a black bobbin winding of 6 x 6 $cm^2$ alternatingly in the vertical direction and horizontal direction at least 6 times to produce a sample. Then,

reflectivity (1%) of light at 540 nm was measured for four given points on the surface of the sample using a Hitachi Automatic Spectrophotometer. Based on the reflectivity, residual pectin is obtained according to the equation shown below.

[0069]

Equation 2

$$\text{Residual pectin (K/S)} = [(1- \text{reflectivity})^2/(2 \times \text{reflectivity})]$$

[0070]

Application Example 7

18 kg of cotton cloth (metsuke 230 g/LY, 20 yarn number count, plain weave) were tossed into 180 L of scouring and bleaching solution consisting of the enzyme composition produced in Application Example 1, 10 mL/L of 35% hydrogen peroxide solution, 2 g/L of Liponox NCI (product of Lion Co., nonionic surfactant), and 0.79 g/L of sodium carbonate and having a pectic acid decomposing activity of the enzyme of 2.9 μ/mL, and reaction was conducted for 20 min at 40°C using a jet dyeing machine; then, treatment was conducted at 95°C for 20 min and the enzyme was deactivated.

[0071]

Application Example 8

10 mL/L of 35% hydrogen peroxide and 0.79 g/L of sodium carbonate were used and the enzyme was deactivated at 95°C for 40 min. The rest of the treatment was done under the conditions described in Application Example 7.

[0072]

Comparative Example 2

Cotton cloth as used in Application Example 7 was dipped in 180 L of scouring and bleaching solution comprised of 8 mL/L of 35% hydrogen peroxide, 1.1 g/L of Gran-Up V0-50K (product of Sanyo Kasei Co., scouring coagent) and 2 g/L of sodium hydroxide and scouring and bleaching were done at 95°C for 30 min using the jet dyeing machine described in Application Example 7.

[0073]

The whiteness, tear strength, tensile strength and residual pectin of the cotton cloth provided with scouring and bleaching in the above-mentioned Application Example 7, Application Example 8 and Comparative Example 2 are shown in Table 6 below.

[0074]

Table 6

| | ② | 綿布の性状 ① | | | | | |
|---|---|---|---|---|---|---|---|
| | | 白色度 | ③引裂強力(g/f) ⑥タテ | ⑦ヨコ | ④張強力(kg/f) ⑥タテ | ⑦ヨコ | ペクチン残存量 (K/S) ⑤ |
| ⑦ | 実施例7 | 92.0 | 2800 | 2360 | >100 | 44 | 1.79 |
| ⑧ | 実施例8 | 92.6 | 3040 | 2560 | >100 | 48 | 1.80 |
| ⑨ | 比較例2 (従来法) | 89.0 | 3000 | 2530 | >100 | 47 | 0.70 |

Key:
1 Properties of cotton cloth
2 Whiteness degree
3 Tear strength (g/f)
4 Tensile strength (kg/f)
5 Residual pectin ratio (K/S)
6 Length
7 Width
8 Application Example ___
9 Comparative Example 2 (conventional method)

[0075]

Each measurement method used in Table 2, Table 3, Table 4 and Table 6 are explained below.

[0076]

① Whiteness degree of cloth

The cloth is cut to form a size of 10 x 10 $cm^2$ to produce a sample. For four given points on the surface of the sample, L, a and b values are measured using a color difference meter CR-200 (product of Minolta Camera Co.) and the whiteness degree is measured based on the values of L, a and b according to the Hunter method using Equation 1 above.

[0077]

② Tear strength

The method specified in JIS L 1096 (Tearing test method for fibers) is used.

[0078]

③ Tensile strength

The method specified in JIS L 1096 (Tensile test method for fibers) is used.

[0079]

④ Residual pectin ratio

The cloth is soaked in 0.02% ruthenium red solution at a bath ratio of 1:40 and vibration is applied for 10 min at 30°C. After vibration, a water wash is provided, vibration is further applied for 30 min at 50°C and a hot-water wash is provided followed by drying; then, the cloth is cut to a size of 10 x 10 $cm^2$ to produce a sample. Then, reflectivity (1%) of light at 540 nm is measured for four given points on the surface of the sample using a Hitachi Automatic Spectrophotometer. Based on the reflectivity, the residual pectin is obtained according to the aforementioned Equation 2.

[0080]

As shown in the results of Table 5 and Table 6, bleaching can be simultaneously done without having an adverse effect on pectin removal in the method of the present invention. Furthermore, the fiber strength is essentially unaffected by the scouring and bleaching process and the whiteness is at a practical level.

[0081]

Effect of the invention

According to the present invention, scouring of natural cellulose type fibers and bleaching of natural cellulose type fibers can be done simultaneously with an enzyme and the process can be simplified, and at the same time, the amount of chemicals used for scouring of the natural cellulose type fibers can be reduced and a pH of wastewater in the range of 6.5 to 7.5 is made possible so wastewater treatment is simplified. And furthermore, the treatment temperature is relatively low and the energy consumption can be reduced compared to that required in conventional methods. And furthermore, the three processes, desizing treatment, scouring treatment, and bleaching treatment, can be simultaneously done.